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Abstract
A preliminary evaluation of several nondestructive testing methods for flaw detection in high-temperature structural ceramic components is being carried out. The ceramics components being investigated include silicon carbide heat-exchanger tubes and silicon nitride rotors. The nondestructive evaluation techniques under consideration include dye-enhanced radiography, holographic interferometry, infrared scanning, acoustic microscopy, acoustic-emission monitoring, acoustic-impact testing, and conventional ultrasonic testing. The capability of each technique to detect critical-size flaws will be discussed. Preliminary results to date' have shown that (a) dye-enhanced radiographic techniques are capable of detecting tight cracks missed with conventional x-ray methods, (b) acoustic microscopy techniques may be useful in detecting and establishing the size of subsurface defects in reaction-bonded silicon nitride, (c) holographic interferometry techniques should be valuable in locating surface cracks in silicon nitride/silicon carbide components, and (d) the results from various silicon carbide tubes suggests that infrared scanning techniques may reveal changes in heat-flow patterns which are related to variations in physical properties. The results for the other techniques mentioned will be discussed. Future efforts in this program, will be directed toward in-depth investigations of the most useful nondestructive techniques.

Keywords
Nondestructive Evaluation

Disciplines
Materials Science and Engineering

PRELIMINARY EVALUATION OF NDE TECHNIQUES FOR STRUCTURAL CERAMICS

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ABSTRACT

A preliminary evaluation of several nondestructive testing methods for flaw detection in high-temperature structural ceramic components is being carried out. The ceramics components being investigated include silicon carbide heat-exchanger tubes and silicon nitride rotors. The nondestructive evaluation techniques under consideration include dye-enhanced radiography, holographic interferometry, infrared scanning, acoustic microscopy, acoustic-emission monitoring, acoustic-impact testing, and conventional ultrasonic testing.

The capability of each technique to detect critical-size flaws will be discussed. Preliminary results to date have shown that (a) dye-enhanced radiographic techniques are capable of detecting tight cracks missed with conventional x-ray methods, (b) acoustic microscopy techniques may be useful in detecting and establishing the size of subsurface defects in reaction-bonded silicon nitride, (c) holographic interferometry techniques should be valuable in locating surface cracks in silicon nitride/silicon carbide components, and (d) the results from various silicon carbide tubes suggests that infrared scanning techniques may reveal changes in heat-flow patterns which are related to variations in physical properties. The results for the other techniques mentioned will be discussed. Future efforts in this program will be directed toward in-depth investigations of the most useful nondestructive techniques.

INTRODUCTION

The objective of this investigation is to establish the feasibility and sensitivity of various NDE techniques for examination of high temperature ceramic components. The techniques under consideration which are discussed here include dye-enhanced radiography, holographic interferometry, acoustic microscopy, acoustic emission, acoustic impact testing and infrared scanning.

DISCUSSION

The first figure shows two silicon nitride rotor blade rings (supplied by Ford Motor Co. for this study) and three silicon carbide heat exchanger tube samples representative of those investigated. The next figure shows schematically the procedure for dye-enhanced radiography where surface flaws filled with an x-ray absorbing dye may be revealed even though missed with conventional radiographic techniques. The third figure shows the mass absorption coefficient ratio for silver nitrate to silicon nitride indicating an absorption edge at 25 KeV. Conventional x-ray machines have a broad spectrum up to a maximum energy value. The optimum setting for a silver nitrate dye appears to be around 50 KeV maximum. Figure 4 shows (for the purpose of illustration) the results using a cracked plastic rod. Figure 6 shows a cross section of a silicon carbide tube (1 mm thick wall) indicating two cracks. The larger crack was detected by both conventional and dye-enhanced radiography; the smaller only by dye-enhanced radiography. Dye-enhanced radiography appears to be useful for detection of tight cracks in ceramic rotors and silicon carbide tubes (particularly for inner wall cracks not accessible with dye-penetrant techniques). Figure 7 shows the schematic arrangement for holographic interferometry. Thermal or mechanical stressing causes visible distortions in holographic interferogram fringe patterns when flaws are present. Figure 8 shows loading modes for ceramic rotor blades. Figure 9 shows the expected fringe distortion for a crack at a blade root. Figure 10 shows examples of interferograms for various samples with mechanical loading. An example of how sensitivities may be enhanced by fringe multiplication techniques is included. Interferograms can be analyzed in a manner shown in Figs. 11 and 12. Surface cracks with characteristic lengths of 750 μm can be detected on the blade root. With special magnification techniques, the resolution may be as small as 100 μ.

Figure 13 describes the equipment arrangement for acoustic microscopy (employing Sonoscan Inc. acoustic microscope). Figures 14 and 15 describe the detection scheme and sample arrangement for ceramic rotor blades (removed from the blade ring). Figure 16 shows a flaw detected in a ceramic rotor blade through acoustic microscopy. This flaw (∿500 x 300 μm) was missed in 3 of 4 radiographs but the presence revealed by the acoustic micrograph was virtually confirmed by metallographic sectioning of the blade (Fig. 17). The acoustic micrographs shown represent an area on the blade 2 x 3 mm. The electronically introduced interference lines are ∿80 μm apart. Acoustic micrographs of SiC heat exchanger tubing show similar

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background structures. Figure 18 shows an acoustic micrograph and visual image of a slice of a ceramic heat exchanger tube (Carborundum αSiC). Surface flaws have been seen both acoustically and optically. Subsurface flaws have also been suggested by acoustic micrographs.

The equipment arrangements and an example for acoustic emission studies are shown in Figures 19-22. The equipment and some data for acoustic impact testing are shown in Figs. 23-26. Figure 27 summarizes the studies for silicon nitride rotors. Figure 28 shows a schematic arrangement for infrared scanning of SiC heat exchanger tubing. The tubes are heated in a water bath and transient patterns observed. Tubes 1, 6 and 7 (counting left to right) are Norton NC430; tubes 2,3,4,5 are Carborundum SiC tubes. Tube 4 is severely cracked. The Norton tubes show better axial heat conduction than the Carborundum tubes. The thermogram is originally in color. In this black and white copy the darker areas are associated with higher temperatures. The cracked tube, as expected shows the worst thermal transport characteristics. A maximum temperature gradient of about 2°C is indicated. Infrared imaging appears to be capable of visually displaying differences in heat transport properties due to variations in physical properties in ceramic tubes and the presence of gross flaws.

Details of the various aspects of this study as well as discussions of conventional ultrasonic testing and fracture mechanics analysis applied to silicon carbide tubing are discussed in references 1-4.

ACKNOWLEDGMENT

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REFERENCES

1. D. S. Kupperman, C. Sciammarella, N. P. Lapinski, A. Sather, D. Yuhas, L. Kessler and N. F. Fiore, "Preliminary Evaluation of Several Nondestructive Evaluation Techniques for Silicon Nitride Gas-Turbine Rotors," Argonne National Laboratory Report ANL-77-89, January, 1978.

2. "Nondestructive Evaluation Techniques for High-Temperature Ceramic Components," Quarterly Report ANL/MSD-78-2, February, 1978.

3. "Nondestructive Evaluation Techniques for High-Temperature Ceramic Components," Quarterly Report ANL/MSD-78-5, March, 1978.

4. Nondestructive Evaluation Techniques for High-Temperature Ceramic Components," Quarterly Report ANL/MSD-78-7, June, 1978.

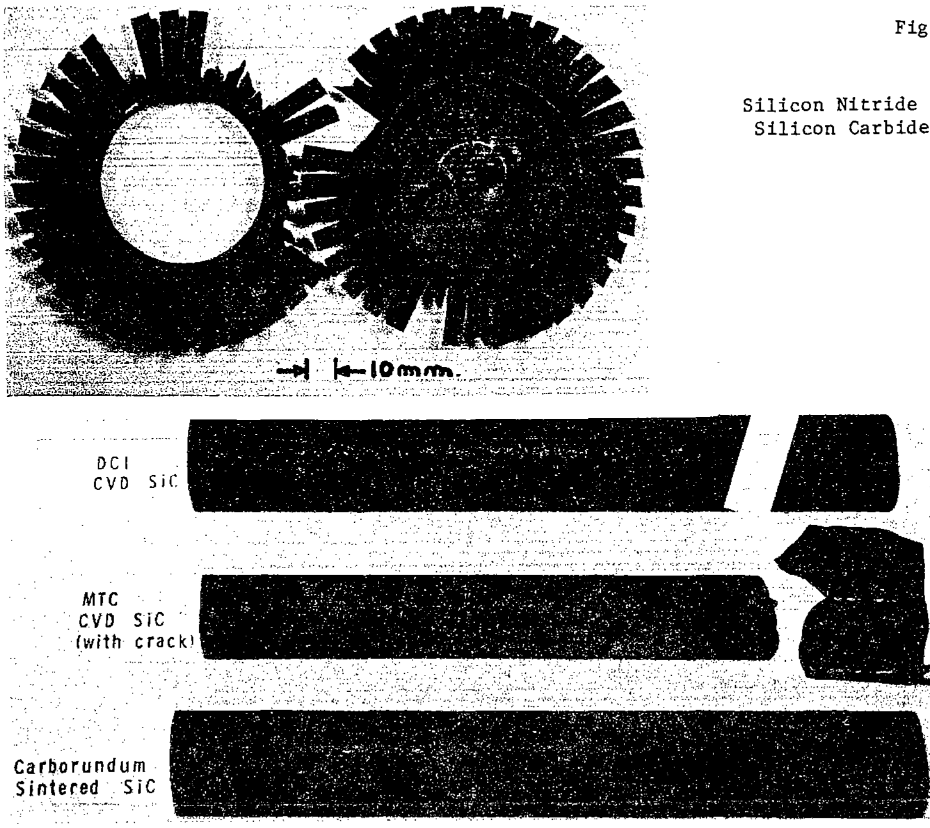


Fig. 1

Silicon Nitride Gas Turbine Rotors and Silicon Carbide Heat Exchanger Tubing

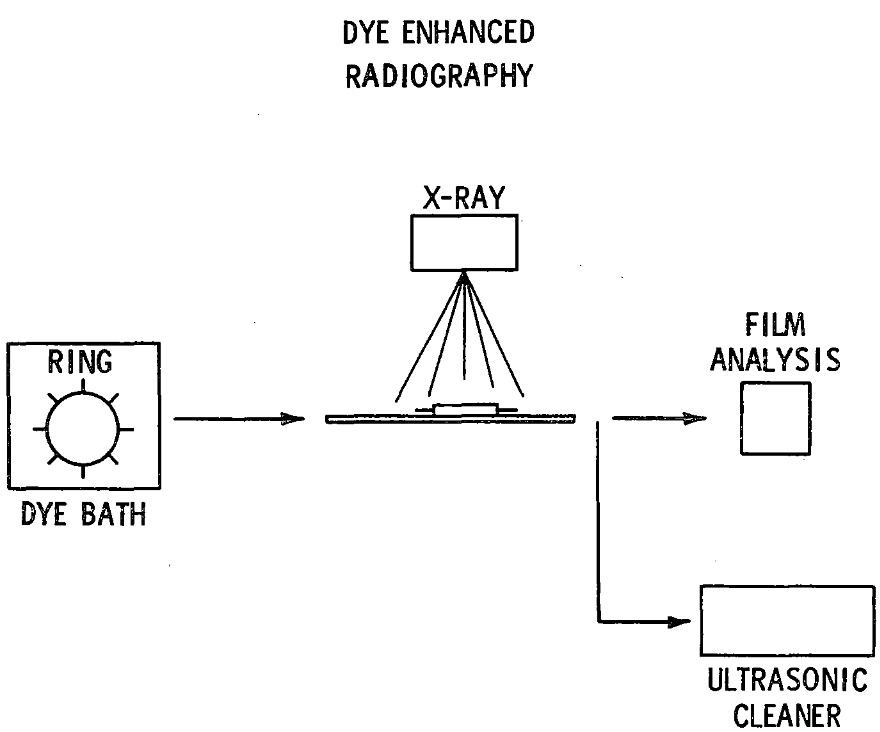


Fig. 2

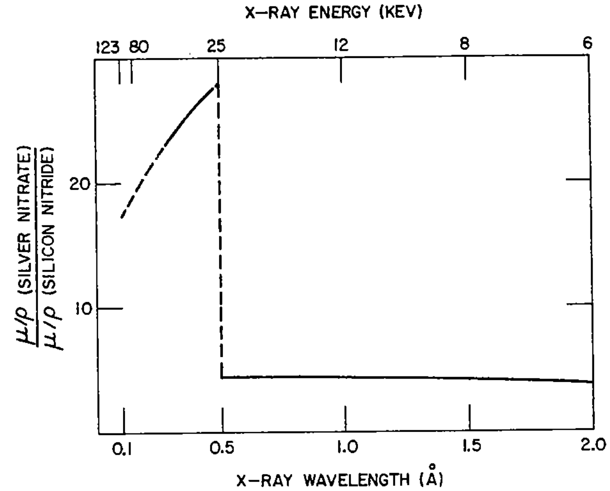


Fig. 3

RADIOGRAPHS OF CRACKED PLEXIGLASS ROD

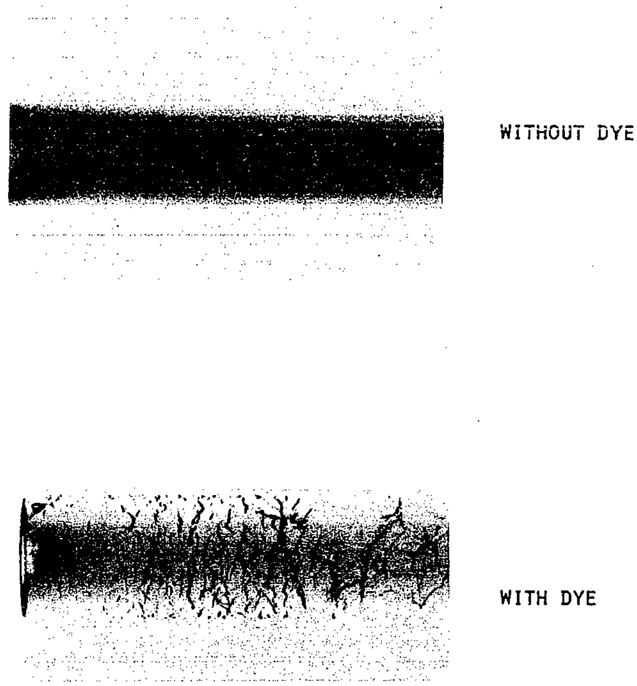
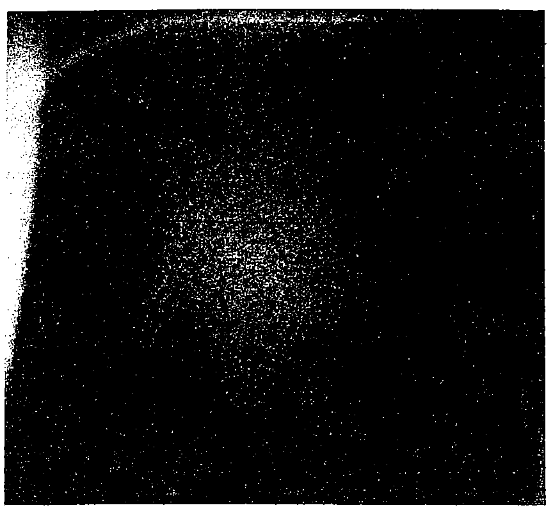


Fig. 4



Anomaly Revealed in Blade-root Region of SN ROTOR. Mag. 20X.

Fig. 5

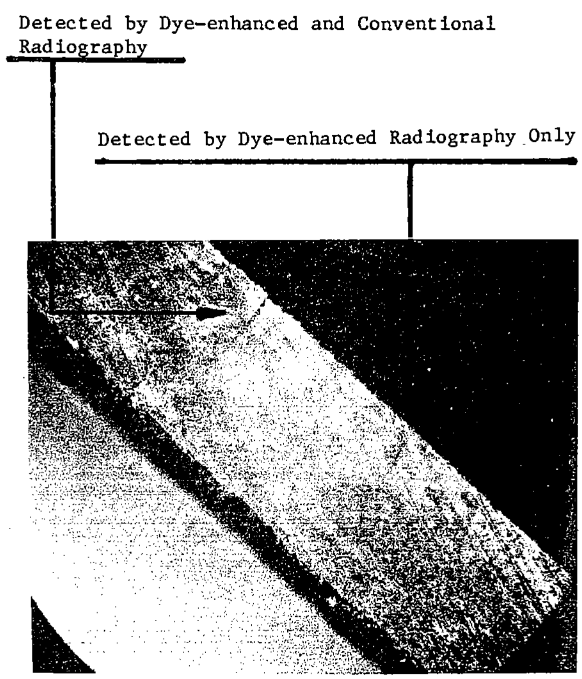


Fig. 6 Cross Section of Ceramic Heat Exchanger Tube, Showing Two Cracks

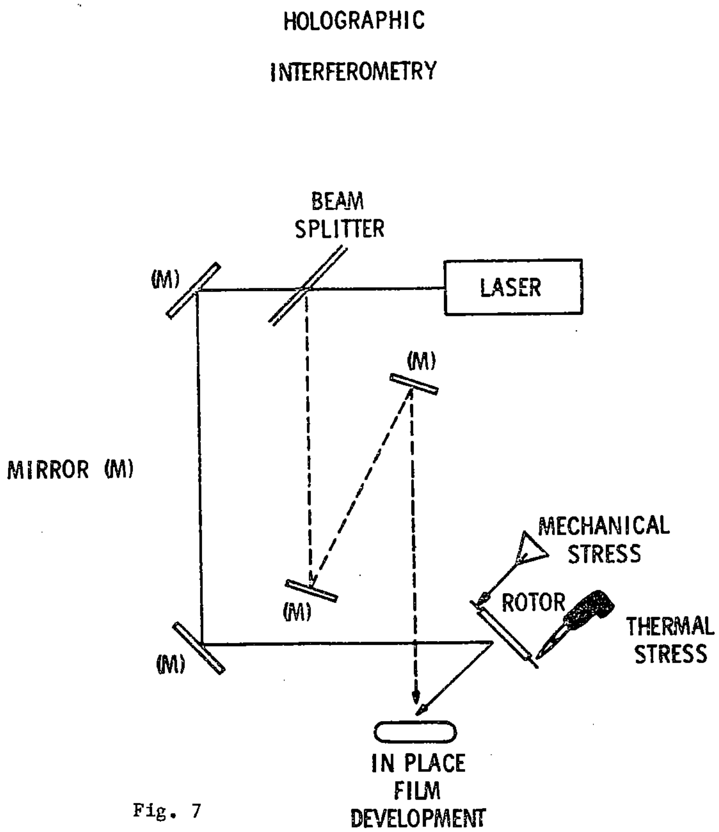


Fig. 7

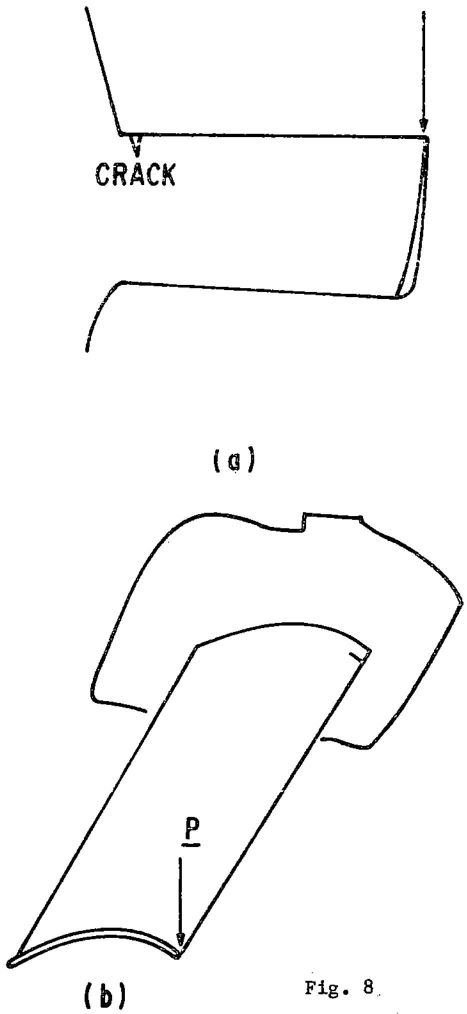


Fig. 8

Modes of Loading of a Turbine Blade

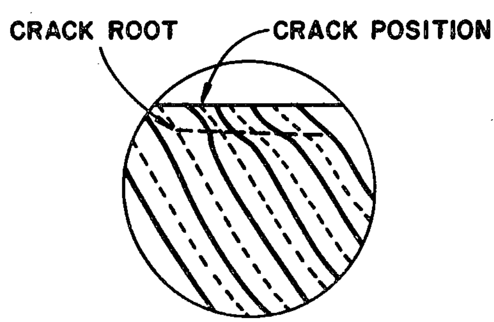
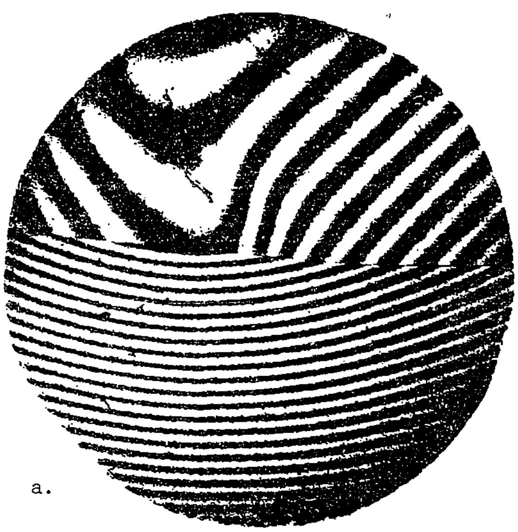
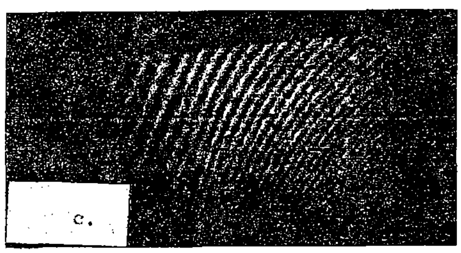


Fig. 9



Holographic Interferograms of (a) Notch in Heat Exchanger Tube; (b) Notch in Plastic Tube; (c) Crack in Rotor Blade; (d) Crack in Plastic disk Showing Fringe Multiplication



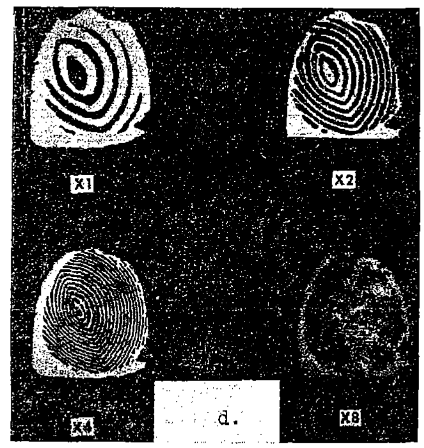


Fig. 10

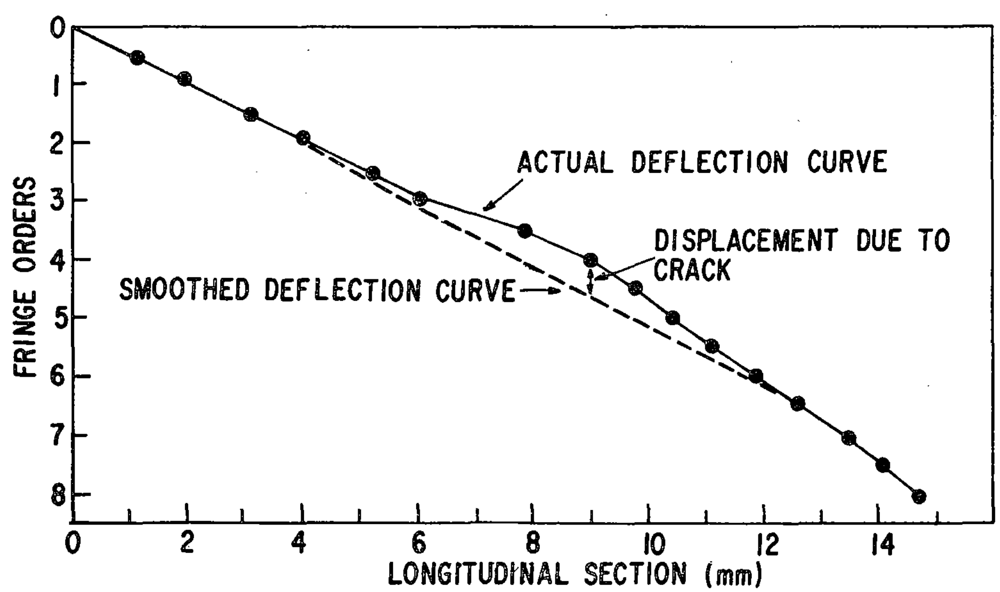
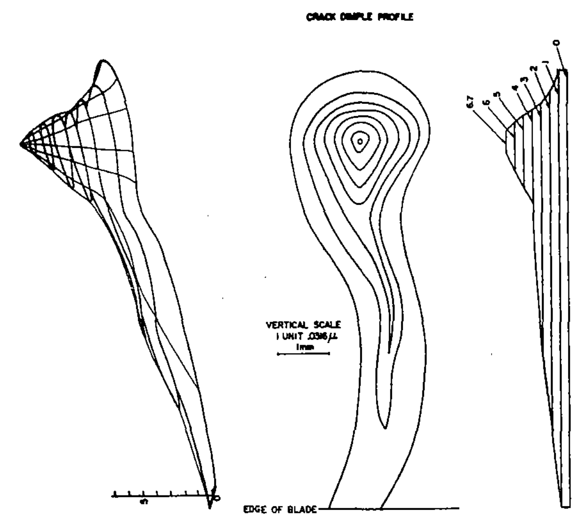


Fig. 11

Plot of Fringe Orders vs Position. By this plot, one obtains the contribution of the displacement field due to the crack from the overall field.



Profile of Dimple Produced by Presence of Crack on Blade

Fig. 12

ACOUSTIC MICROSCOPY

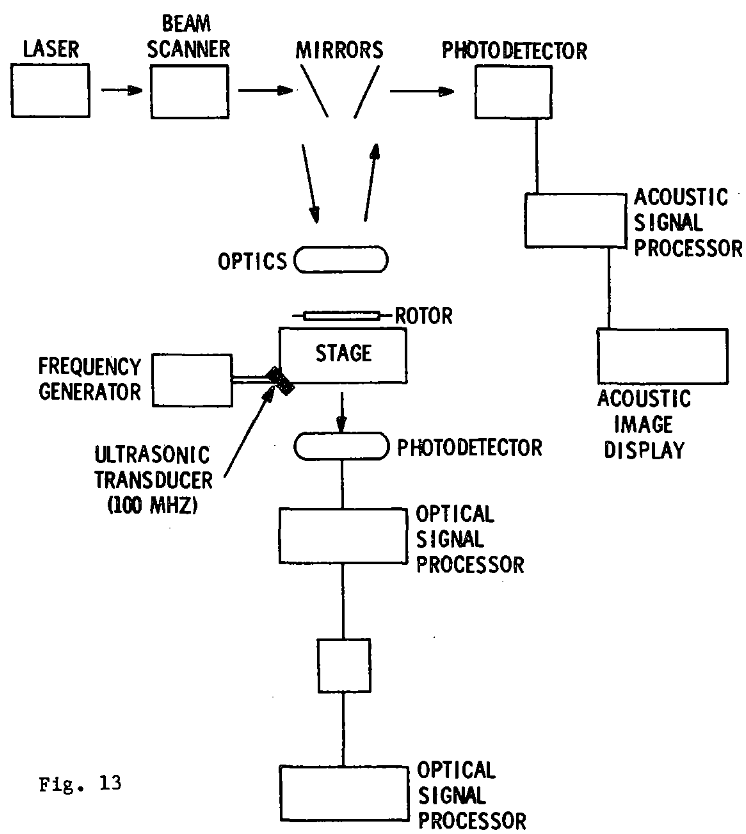
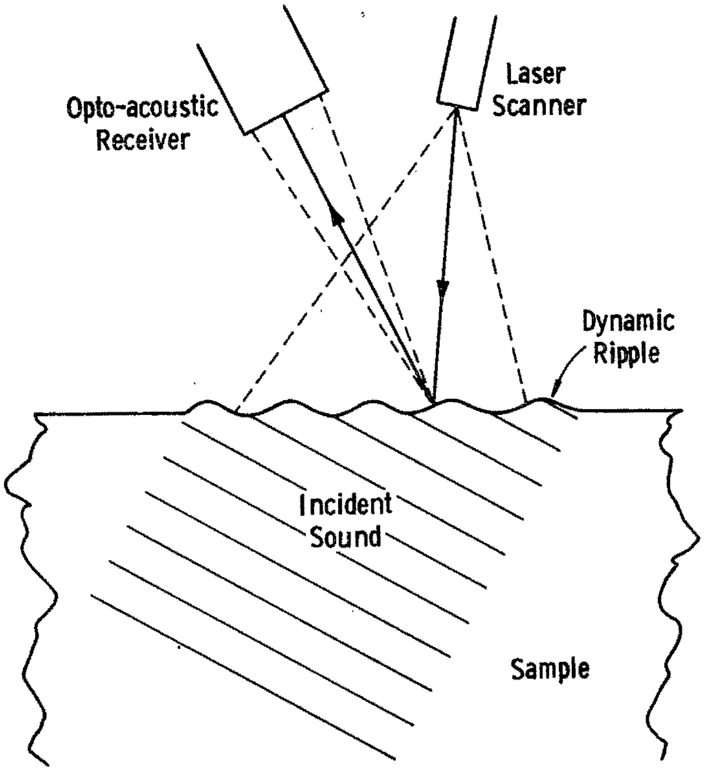
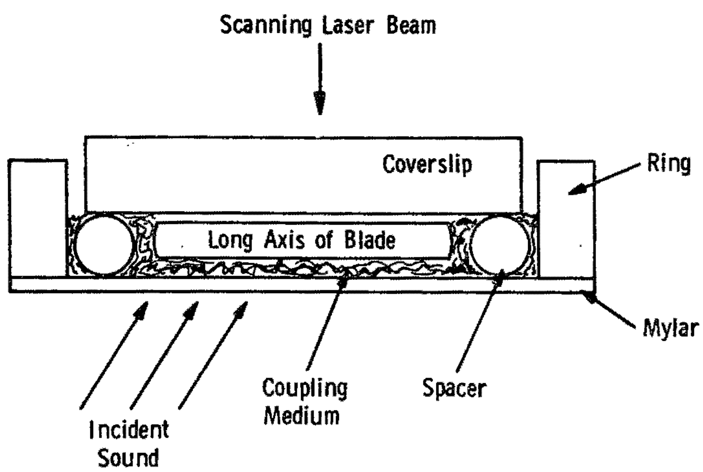


Fig. 13



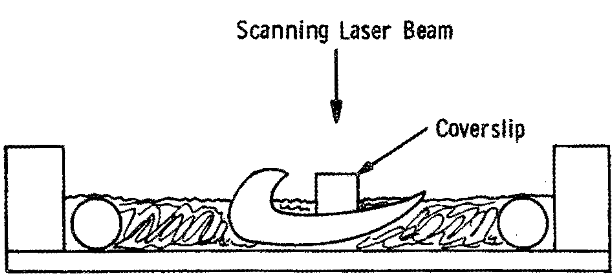
Schematic Diagram Illustrating Detection Scheme Used by Scanning Laser Acoustic Microscope

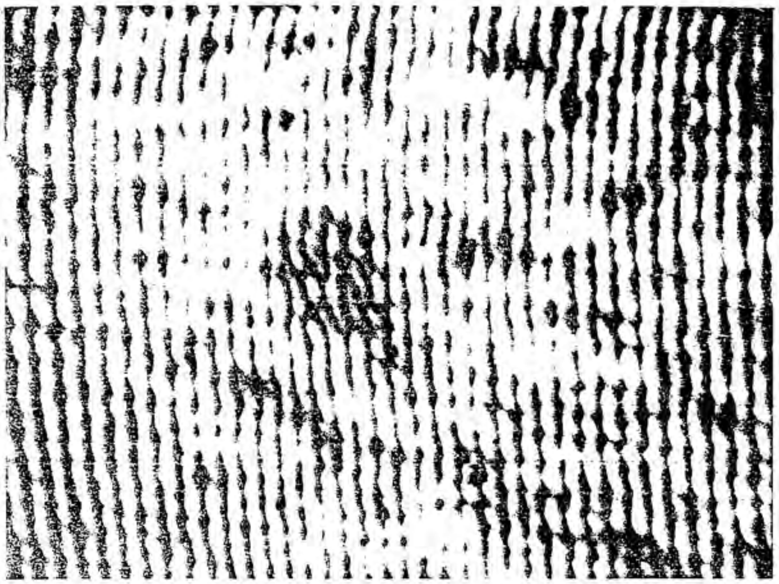
Fig. 14



Schematic Diagram of Sample Configuration

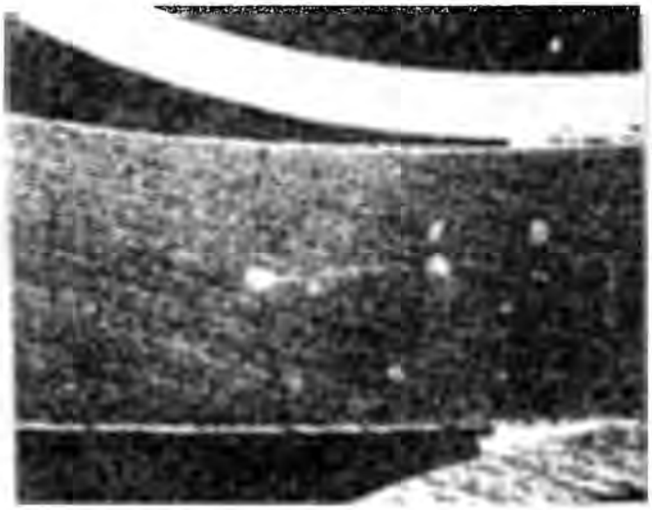
Fig. 15





Defect Cluster (circled) Observed in Flatter Portions of Blade 28

Fig. 16

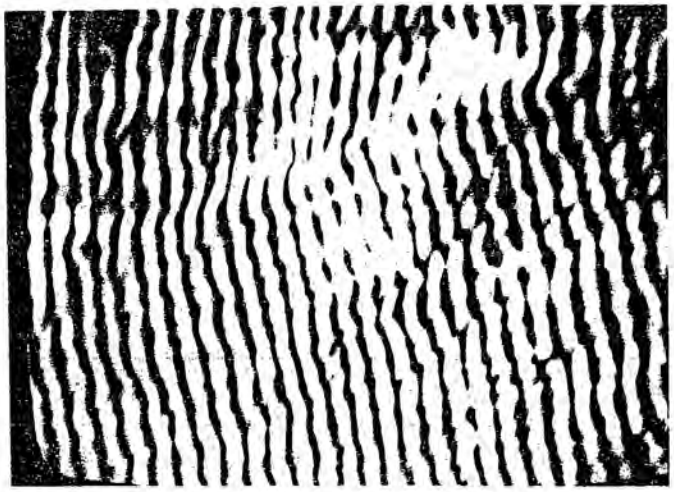


Cross Section of Blade at Location of Pore Indicated by Radiography and in General Region of Defect Indicated by Acoustic Microscopy

Fig. 17

Acoustic Micrograph of Section of Silicon Carbide Heat Exchanger Tubing

Fig. 18



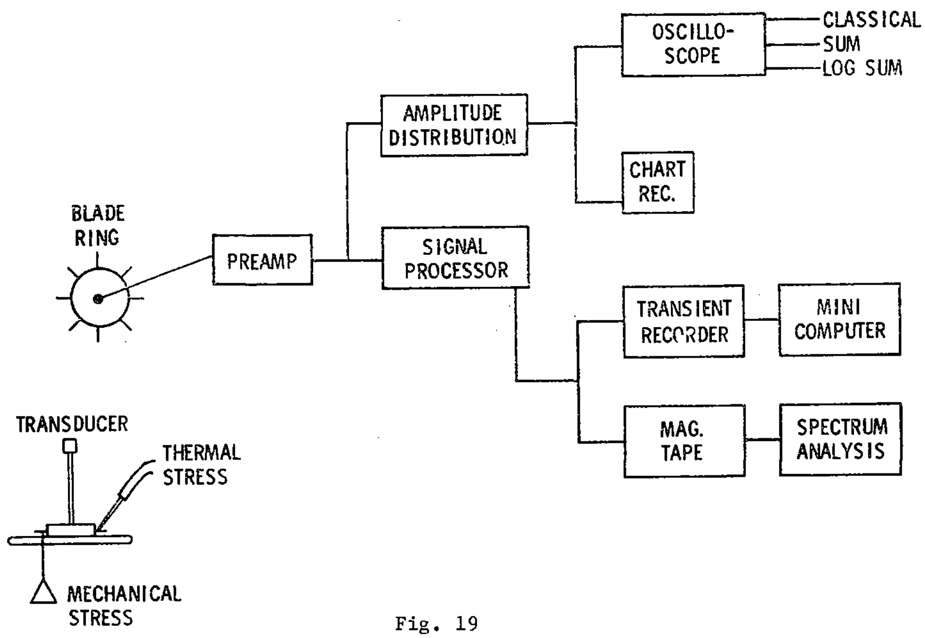
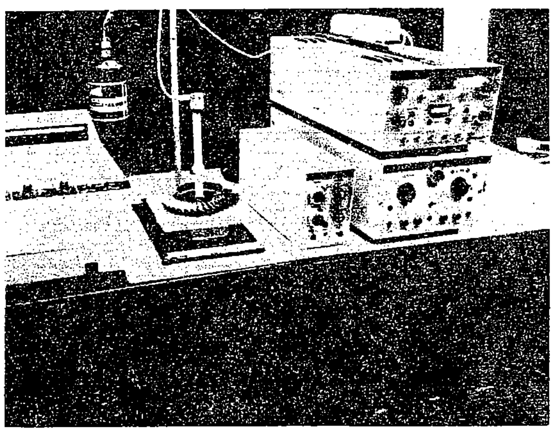


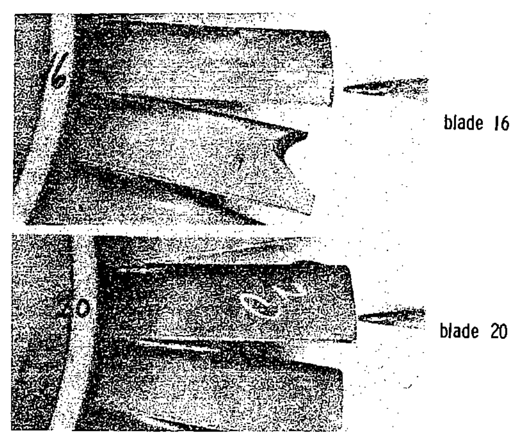
Fig. 19



Photograph of SN Rotor, SN Stand, Amplitude-distribution Analyzer, and Signal Processor for Acoustic-emission (AE) Experiments

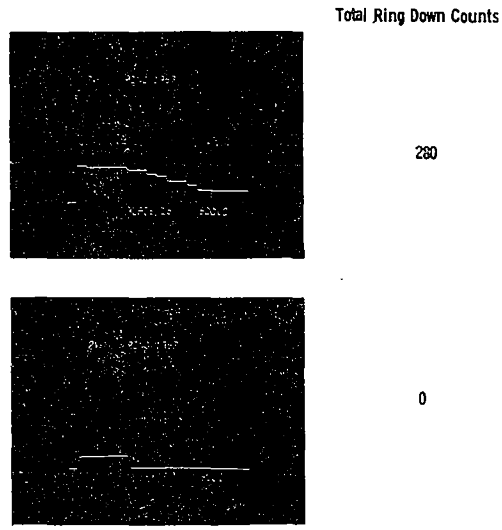
Fig. 20

BLADE RING 1957



Blades 16 and 20 of Blade Ring 1957

Fig. 21



Amplitude-distribution-data Cumulative Log and Total Counts for Thermal Stressing of Blades 16 and 20 of Ring 1957.

Fig. 22

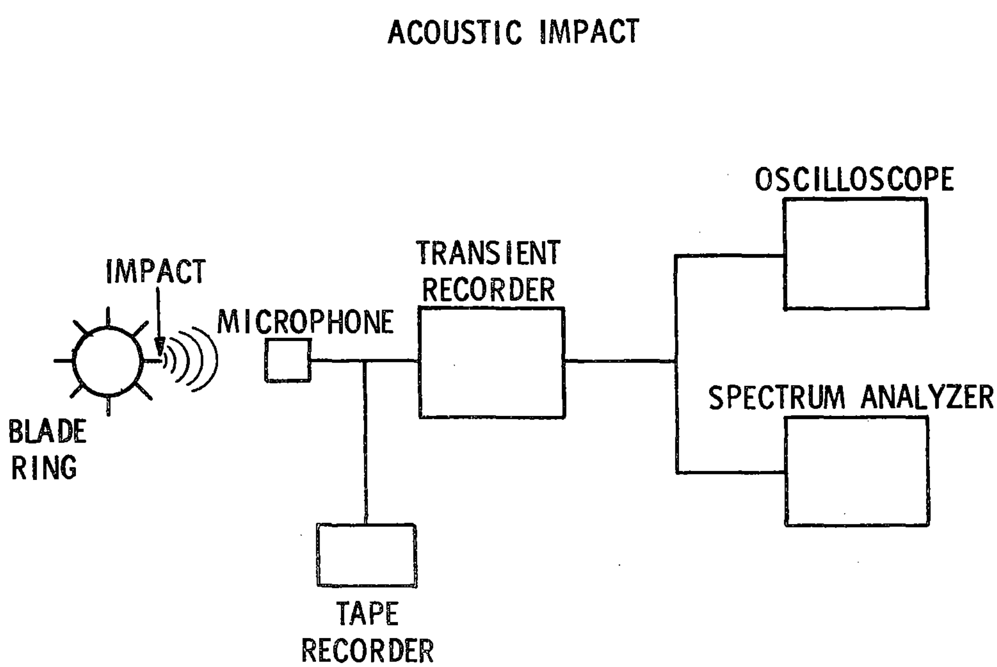


Fig. 23

RESONANCE FREQUENCY FOR ROTOR BLADE IN CANTILEVER MODE OF VIBRATION

$$f = \frac{A}{2\pi} \sqrt{\frac{E b^2}{12 \rho \ell^4}}$$

$E = 300 \times 10^{10}$ DYNES/CM^2 (MODULUS OF ELASTICITY)

$\rho = 2.7$ G/CM^3 (DENSITY)

$b = 0.2$ CM (AVERAGE THICKNESS OF BLADE TAPER)

$\ell = 2.5$ CM (BLADE LENGTH)

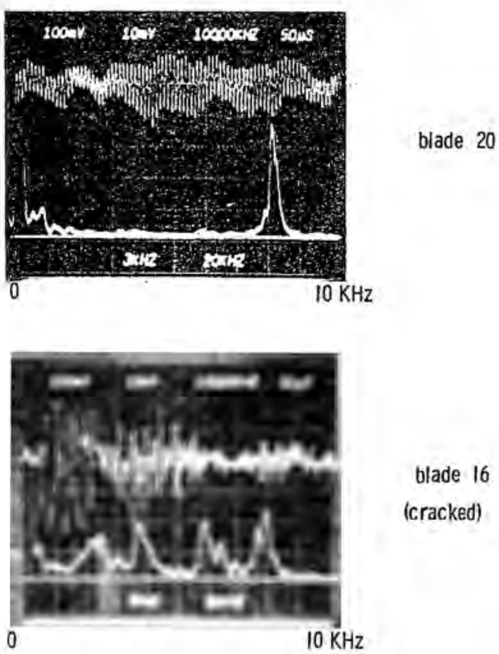
$A = 11.7$ (GEOMETRIC FACTOR FOR FUNDAMENTAL MODE WITH TAPERED 3b TO b)

$f_{CALC} = 9.1$ KHz

$f_{MEASURED} = 8$ KHz

Fig. 24

ACOUSTIC IMPACT TESTING

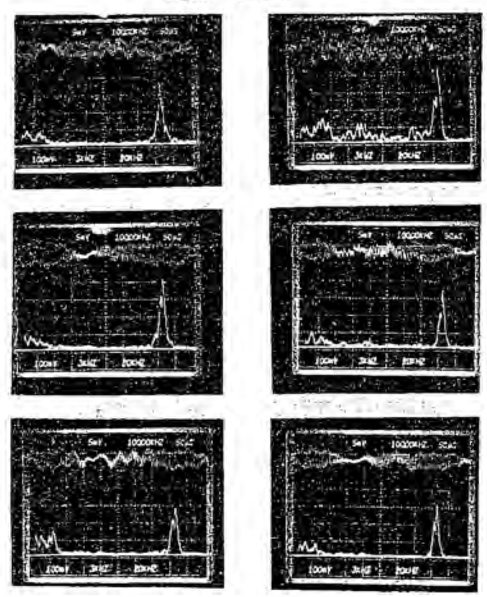


Frequency Spectra of Blades 16 and 20 of Blade Ring 1957, Indicating Variation in Blade Quality. Blade 16 has a tight radial crack from the blade tip to about midway between the blade tip and blade root.

Fig. 25

ACOUSTIC IMPACT TESTING

Ring 2319 Blade 12



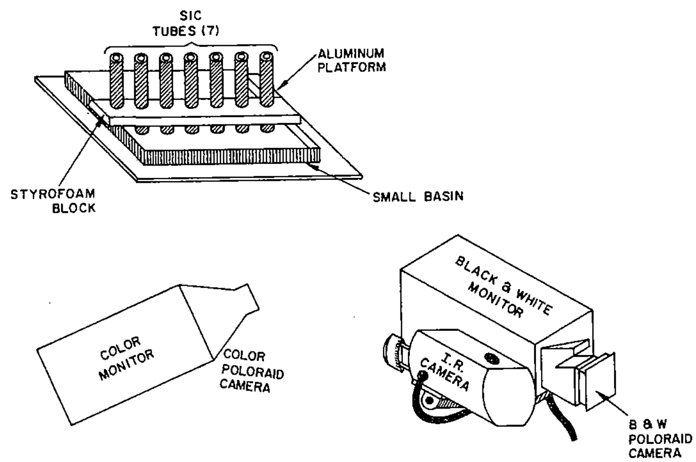
Six Consecutive Impacts (Using pencil-lead technique) and Resulting Frequency Spectrum from Blade 12 of Blade Ring 2319.

Fig. 26

NDE TECHNIQUES FOR SILICON NITRIDE ROTORS

METHOD	ADAPTABILITY TO ROTOR GEOMETRY	FLAW DETECTION		LEVEL OF DIFFICULTY TO DETECT CRITICALLY SIZED FLAWS	COMMENTS
		SURFACE	SUBSURFACE		
1) DYE ENHANCED RADIOGRAPHY	EXCELLENT	X		MODERATELY LOW	CAN REVEAL FLAWS NOT VISIBLE BY ORDINARY X-RAY OPTICAL OR DYE PENETRANT METHODS. VERY PROMISING
2) ACOUSTIC MICROSCOPY	FAIR	X	X	LOW	OVERALL QUALITY OF COMPONENT AS WELL AS SPECIFIC DEFECTS CAN BE OBSERVED. LIMITED TO 3 MM THICK SPECIMEN IN RB, 6 MM THICK SPECIMEN IN HP.
3) HOLOGRAPHIC INTERFEROMETRY	GOOD	X	?	MODERATE	PROBABLY ADAPTABLE TO AUTOMATIC SCANNING
4) ACOUSTIC EMISSION	FAIR	X	X	HIGH	DATA INTERPRETATION VERY DIFFICULT, RELIES ON FLAW POPULATION CHANGE DURING TEST TO RELEASE ACOUSTIC ENERGY FOR FLAW INDICATION
5) ACOUSTIC IMPACT TESTING	GOOD	X	X	HIGH	INDICATE OVERALL COMPONENT QUALITY
6) INTERNAL FRICTION	POOR	X	X	HIGH	PARTICULARLY DIFFICULT TO ADAPT TO ROTOR INSPECTION

Fig. 27



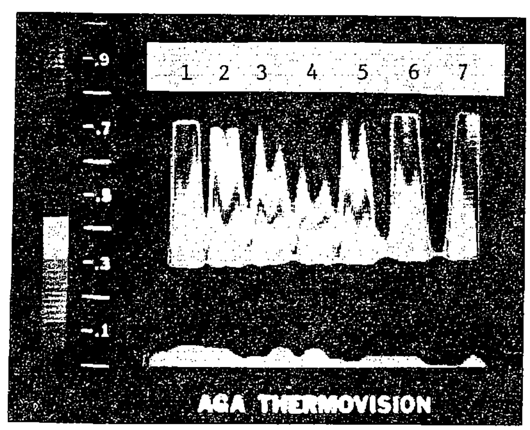


Fig. 28

Thermogram of Heat Exchanger Tubing

Tubes 1, 6, 7 are Norton Co.

Tubes 2, 3, 4, 5 are Carborundum

Tube 4 is cracked